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Q-Switch Laser and Tattoo Pigments: First Results of the Chemical and Photophysical Analysis of 41 Compounds

Wolfgang Bäumler, PhD,[1]* Ernst T. Eibler, PhD,[2] Ulrich Hohenleutner, MD,[1] Benno Sens, PhD[3] Jürgen Sauer, PhD,[2] and Michael Landthaler, MD,[1]

[1]Department of Dermatology University of Regensburg, Germany
[2]Department of Organic Chemistry, University of Regensburg, Germany
[3]BASF AG, Germany

Background and Objective: **In the Western world, there are at least 20–30 million people with tattoos. Improved self-image and social stigmatization are the main reasons for removing tattoos from skin. Q-switched lasers are applied to destroy the tattoo compounds in the skin. The treatment of tattoos containing ink often gives excellent results, whereas the results of treatments for coloured tattoos are not predictable and usually are worse. The chemical structure and the absorption spectra of the tattoo pigments are usually unknown. However, the efficacy of the treatment by using light of different Q-switched lasers (wavelengths 510, 532, 694, 755, 1064 nm) is correlated to both the chemical structure of the tattooed compounds yielding specific absorption spectra and the laser wavelength used.**
Study Design/Materials and Methods: **A structural and spectroscopic analysis of 41 coloured pigments was performed.**
Results: **The 41 substances were identified, and they consist of 16 individual chemicals of different structured well-known industrial organic pigments. The absorption spectra of the 16 pigments were measured quantitatively.**
Conclusion: **The results of the present analysis explain to some extent the outcome of clinical studies regarding laser therapy of coloured tattoos. Because the laser energy used produces a high temperature in the azo or polycyclic pigments, it is necessary to investigate whether that change causes possibly toxic or cancerogenic compounds. Lasers Surg. Med. 26:13–21, 2000.** **© 2000 Wiley-Liss, Inc.**

Key words: absorption spectra; cancerogenicity; chemical structure; coloured tattoo; Q-switched laser; toxicology

INTRODUCTION

Today tattooing in the Western world is considered to be a sign of self-destructive and rebellious behaviour [1]. Kilmer et al. [2] estimated that approximately 10 million people in the United States have at least one decorative tattoo. Cosmetic tattoos, in which black, red, or flesh-toned pigments are used to mimic eye, lip or eyebrow liner, have also become increasingly popular [2].

In the past, colouring agents were inorganic pigments such as titanium dioxide (TiO_2, white), cadmium sulphide (CdS, yellow), chromic oxide (Cr_2O_3, green), cadmium selenide (CdSe, red), red cinnabar (HgS, red), iron oxide (Fe_2O_3, red, Fe_3O_4, black), and carbon (black) [3]. For dark-blue amateur tattoos, commercially available ink is still in use.

Professional tattoos have different colours and consist of a variety of pigments and neither the tattoo artist nor the tattooed patient has any information about the compounds punctured into skin. The tattoo compounds (powder or emul-

*Correspondence to: Dr. Wolfgang Bäumler, Department of Dermatology, University of Regensburg, 93042 Regensburg, Germany.
E-mail: baeumler.wolfgang@klinik.uni-regensburg.de

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sions) are sold to the tattoo manufacturer by a few tattoo suppliers. Because tattoo compounds in comparison with cosmetics are not officially controlled, the origin and chemical structure of these colouring agents are hardly known.

Organic pigments are classified by their chemical constitution [4]. A rough distinction is made between azo and non-azo (polycyclic) pigments. Generally, the pigments used for tattooing are well tolerated by the skin. Nevertheless, adverse reactions have been published in the literature, for instance allergic reactions [5–10], pseudolymphomas [11–13], systemic sarcoidosis [14,15], and granulomatous or lichenoid reactions [16,17]. In addition, several malignant lesions have occurred in tattoos (possibly coincidental), including basal cell carcinomas [18], squamous cell carcinomas [19], and malignant melanomas [20,21]. In one case, an evolution of B-cell lymphoma from pseudolymphoma has been reported [22]. Allergic reactions are induced by mercury ions, chromium III ions, or cobalt II ions. Cadmium sulphide and cadium selenide are mainly responsible for phototoxic reactions.

In addition to these adverse reactions, the main reasons for removing tattoos are improved self-image or social stigmatization. Traditional modalities are the removal of the pigment-containing skin by using salabrasion [23,24], cryosurgery [25,26], surgical excision [27,28] or CO_2-laser application [29,30]. However, these methods induce permanent scarring. Selective photothermolysis has been published [31] for the treatment of vascular lesions. The tattoo pigments have smaller diameters as compared with the vessels. Thus, the application of laser pulses with a high-intensity and an ultrashort pulse duration of a few nanoseconds is necessary when using Q-switched lasers. In addition, the laser should emit light at a wavelength that is well absorbed by the pigments. Until now, the laser systems used have fixed wavelengths such as pulsed dye (510 nm), ruby (694 nm), Nd:YAG (532 or 1064), and alexandrite (755 nm) [32–36]. It is quite uncertain, to what an extent tattoo pigments would absorb these wavelengths, only clinical studies are present. [37]. In addition to the interaction with tattoo pigments, ultrashort pulses also destroy melanosomes in the skin, which are similar in size and cellular location as tattoo pigments.

To date, only a few data regarding the chemical structure and the optical absorption of tattoo pigments have been published in the literature [3]. Consequently, a goal of the present investigation was the chemical analysis of the pigments, as well as the measurement of the ultraviolet-visible-near infrared (UV-VIS-NIR) absorption spectra.

MATERIAL AND METHODS

Tattoo Pigments

A thorough market analysis carried out by representatively consulted tattoo manufacturers and their suppliers revealed that in the Western world, there are only a few wholesalers from the United States, the United Kingdom, The Netherlands, and Germany. They are mentioned here as company 1, 2, 3, 4, or 5. From a variety of tattoo compounds, 41 specimens representatively were selected and analyzed consisting of blue (5), green (9), yellow (6), orange (5), red (12), and violet (4) pigments.

Absorption Spectra

Absorbance spectra $\varepsilon(\lambda)$ (320–1100 nm) of the pigments in commercial quality were determined independently by two spectrophotometers (Specord M500, Carl Zeiss GmbH, Jena, Germany, and Lambda 2, Perkin Elmer, Überlingen, Germany) by using quartz cells with 10-mm lengths. Pigment (0.06–0.6 mg) was dissolved in 25 ml of appropriate solvents: 1,2,4-trichlorobenzene (TCB, LiChrosolv quality, Merck KGaA, Darmstadt, Germany), a mixture of TCB (80 ml) and phenol (20.0 g, Merck, p.a.), a mixture of TCB (80 ml) and phenol (30.0 g), chloroform (LiChrosolv, Merck), pyridine (p.a. >99.5 %, dried, Merck).

Spectroscopy

To identify the unknown compounds, infrared (IR) spectroscopy was used as fingerprinting. FTIR spectra (4,000–400 cm^{-1}, 4 cm^{-1} resolution) were performed by using a 510M IR-interferometer (Nicolet Instrument GmbH, Offenbach, Germany), KBr discs (pigment concentration 0.1% w/w), and background correction. Pigments were identified by computer-assisted pattern comparison with original pigment spectra from data banks.

Electron Microscopy

The pigments were suspended in ethanol and applied to a microscope slide by ultrasound vaporization. Transmission electron microscopy

(TEM) was used to evaluate the size and the shape of the pigments.

X-Ray Powder Diffraction

X-ray powder diffraction data of the pigment samples, supported by existing data bases with reference substances, served as an additional and independent method to support the used IR identification. The measurements were performed by using a diffractometer D5000 (theta, 40 kV, CuK_{α}, Siemens AG, Germany).

Mass Spectrometry

Mass spectrometry was done in PI-EI mode (70 eV) with DIP inlet (tungsten lamp) on a MAT 95 spectrometer (Finnigan-MAT GmbH, Bremen, Germany).

RESULTS

Chemical Classification of the Analyzed Pigments

The analysis of the pigments by using standard methods turned out to be very troublesome because of their extremely low solubility or volatility. In addition, insoluble inorganic admixtures such as TiO_2 or $BaSO_4$ are used to lighten the original colour. Nevertheless, we were able to identify all analyzed pigments and out of 41 tattoo colour preparations we selected 16 real chemical individuals.

The results of identification of the coloured pigments by using IR-spectroscopy, X-ray powder diffraction, and mass spectroscopy are listed in Tables 1 (azo compounds) and 2 (polycyclic compounds) containing the pigment number, the colour index (CI), the colour, and the trade name of the pigment. The last two columns of Tables 1 and 2 include the chemical classification of the pigments and the chemical structure, the basic structural formula of the tattoo pigments, and the different substituents are shown in Figure 1.

Although there are different colours and trade names, only 16 pigments are real chemical individuals. For example, the green pigment P.G. 7 is marketed by five different tattoo suppliers by using seven different trade names.

Absorption Spectra

The absorption spectra of the pigments are shown in Figure 2A–C. The polycyclic pigments were strongly absorbing in the red part of the visible spectrum, whereas the azo pigments had an absorption maximum in the blue-green part. The values for the absorption maximum are ranging from 0.85 (P.R. 122) to 6.7 (P.Y. 83) (molar extinction ε 10^{-4} mol^{-1} cm^{-1}). The symbols in Figure 2A–C indicate the wavelengths of the Q-switched lasers used. The yellow pigments P.Y. 14 and P.Y. 74 exhibit no absorption for all laser wavelengths used. The ruby-, Nd:YAG-, and alexandrite laser (694, 755, 1,064 nm, respectively) hardly match the absorption spectra of the analyzed yellow or red pigments. The absorption values of polycyclic pigments are quite different regarding the laser wavelength used (i.e., 510, 532, 694, 755 nm).

Electron Microscopy

The TEM pictures of the pigments showed a variety of shapes such as needles, platelets, cubes, bars, and a number of irregular shapes. Besides primary particles, aggregates composed of primary particles grown together at their surfaces, and agglomerates (groups of single crystals joined together at their edges) are present in the same picture. The diameters of the pigments vary from about 20 nm to 900 nm. In Figure 3, two different shapes corresponding to two different tattoo suppliers of the pigment P.R. 122 are demonstrated.

DISCUSSION

Tables 1 and 2 show that most of the tattoo compounds analyzed were industrial organic pigments, and 16 individual chemicals are sold by using 41 different trade names. In view of the variety of tattoo pigments on the market, our list of analyzed tattoo pigments is definitely incomplete, but only one-third of the purchased pigments are individual chemicals according to our classification. The absorption spectra provide evidence of the high colour strength of the pure pigments such as P.G. 7. Therefore, the pigments are often mixed with different amounts of titanium dioxide to lighten the basic colour. These mixtures have their own trade names, and the number of tattoo compounds increases without increasing the number of basic pigments, which are the target of tattoo removal by using Q-switched lasers. There are only a few publications dealing with the chemical structure of tattoo pigments. In 1988, Lehmann et al. [3] investigated seven coloured pigments used for tattooing. The authors also identified, according to our classification, P.Y. 74, P.Y. 83, P.R. 22, and two Cu-phthalocyanines, one yellow diarylide pigment (P.O. 16) and one violet quinacridone pigment (P.V. 19).

For the interaction of pigments and Q-

TABLE 1. Azo Compounds

No.	Pigment[a]	Colour	Trade name[b] (distributor)		Chemical structure[c]	
1	P.Y. 14 C.I. 21095 CAS No. 5468-75-7	Yellow	Canary Yellow*§#	(2)	Disazo-diarylide	(*1a*)
2	P.Y. 55[d] C.I. 21096 CAS No. 6358-37-8	Reddish yellow	17#	(3)	Disazo-diarylide	(*1b*)
3	P.Y. 74 C.I. 11741 CAS No. 6358-31-2	Greenish yellow	Zitronengelb§#	(1)	Monoazo-pigment	(*2*)
4	P.Y. 83 C.I. 21108 CAS No. 5567-15-7	Reddish yellow	Dunkelgelb# Golden Luv*	(1) (2)	Disazo-diarylide	(*1c*)
5	P.Y. 87[e] C.I. 21107:1 CAS No. 15110-84-6	Reddish yellow	Sunset Yellow#	(4)	Disazo-diarylide	(*1d*)
6	P.0. 13 C.I. 21110 CAS No. 3520-72-7	Yellowish orange	Orange Navel Orange# Melon* I3 [P7]*	(1) (2) (2) (3) (5)	Disazopyrazolone	(*1e*)
7	P.R. 5 C.I. 12490 CAS No. 6410-41-9	Carmine	Dunkelrot#§	(2)	NAS-Pigment	(*3a*)
8	P.R. 9 C.I. 12460 CAS No. 6410-38-4	Yellowish red	I8#	(3)	NAS-Pigment	(*3b*)
9	P.R. 22 C.I. 12315 CAS No. 6448-95-9	Yellowish red	Cardinal Red# Dragon Red Spanish Red [P8][f]	(2) (4) (4)	NAS-Pigment	(*3c*)
10	P.R. 112 C.I. 12370 CAS No. 6535-46-2	Red	Ruby Red*§ Red Velvet§#	(2) (2)	NAS-Pigment	(*3d*)
11	P.R. 170 C.I. 12475 CAS No. 2786-76-7	Red	[P1]#	(5)	NAS-Pigment	(*3e*)

[a]Colour index pigment name and constitution number. CAS No. refers to the CAS registry number (Chemical Abstract Service of the American Chemical Society).
[b]An asterisk refers to pigment samples diluted by TiO_2 (mostly as anatase, but in Melon (2), Dunkelgelb (1), and Sunset Yellow (4) as rutile). All pigment samples (without I7 (3) and Sunset Yellow) were identified by database-supported infrared spectrometry. The structures of substances marked by section symbols and pound signs were in addition elucidated by X-ray analysis and mass spectrometry, respectively. Numbers set in brackets are substitutes for unknown trade names of pigments purchased directly from local tattooists.
[c]See Figure 1.
[d]The compound was analyzed by mass spectroscopy only.
[e]The chemical isomer P.Y. 124 cannot be excluded reliably, the methoxy groups in position R^3 and R^4 in Figure 1 would then change to position R^2 and R^5, respectively.
[f]Patient sample; trade name, distributor, and tattooist are unknown.

switched lasers, it is necessary that the wavelength of the laser light has to match the absorption spectra of the tattooed compounds. The results of our investigations demonstrate that not all laser wavelengths (510, 532, 694, 755, and 1064 nm) used are well absorbed in the various pigments. Moreover, tattoos having similar colours may contain completely different pigments and, therefore, exhibit a different absorption behaviour. The light pulses from the Q-switched ruby laser (694 nm) are well absorbed in the blue-green pigments P.B. 15 and P.G. 7. This finding is in agreement with the therapeutic outcome of Kilmer and Anderson [2] and Levine and Geronemus. [34]. They reported that green and blue tattoos had a better response to ruby laser compared with the Nd:YAG laser. Ferguson and August [33] found that yellow, orange, and green tattoos did

TABLE 2. Polycyclic Compounds

No.	Pigment[a]	Colour	Trade name[b] (distributor)		Chemical structure[c]	
12	P.R. 122	Bluish red	Burgandy*§	(2)	Quinacridone	(*4*)
	C.I. 73915	(magenta)	I5§#	(3)		
	CAS No. 980-26-7		Magenta*	(1)		
13	P.V. 23	Bluish violet	I6	(3)	Dioxazine[d]	(*5*)
	C.I. 51319		Pur Purple#	(1)		
	CAS No. 6358-30-1		True Purple*§	(2)		
			[P3]*#	(5)		
14	P.B. 15	Blue	Permanentblau‡§	(1,α)	Cu-	(*6*)
	C.I. 74160		Fezan Lt. Blue§	(4,α)	Phthalocyanine	
	CAS No. 147-14-8		I1§	(3,β)	(α,β-modification)	
			Permanent Blue§#	(2,β)		
			Navy Blue*§	(2,β)		
15	P.G. 7	Bluish green	Permanentgrün‡§	(1)	Cu-	(*7*)
	C.I. 74260		Waldgrün*	(1)	Hexadecachloro-	
	CAS No. 1328-53-6		Forest Green§	(2)	phthalocyanine	
			I4§	(3)		
			Fezan Blue Green§	(4)		
			Permanent Green§	(4)		
			[P2]*#	(5)		
16	Polymon Green	Green	Avocado Green*	(2)	Cu/Al-	(*8*)
			E3	(3)	Phthalocyanin- Br_xCl_y	

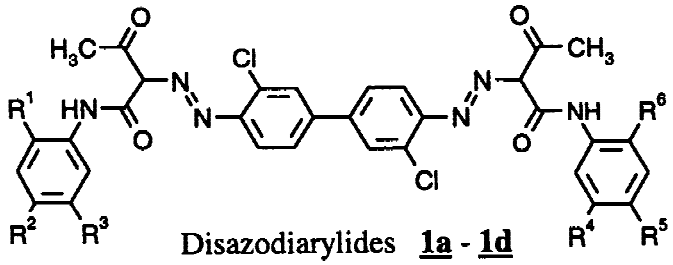
[a]Colour index pigment name and constitution number. CAS No. refers to the CAS registry number (Chemical Abstract Service of the American Chemical Society).
[b]An asterisk refers to pigment samples diluted by TiO_2 (mostly as anatase, but in Melon (2), Dunkelgelb (1), and Sunset Yellow (4) as rutile). The double-dagger symbol reveals $BaSO_4$ (barite) as an additive. All pigment samples (without I7 (3) and Sunset Yellow) were identified by database-supported infrared spectrometry. The structures of substances marked by section symbols and pound signs were in addition elucidated by X-ray analysis and mass spectrometry, respectively. Numbers set in brackets are substitutes for unknown trade names of pigments puchased directly from local tattooists.
[c]See Figure 2.
[d]The exact structure still seems to be controversial.

not respond to therapy by using the Nd:YAG laser at 532 or 1064 nm. These findings correlate to the low absorption of the pigments P.Y. 74 (yellow), P.O. 13 (orange), and P.G. 7 (green) at 532 nm. However, the light from the Q-switched and frequency doubled Nd:YAG laser (532 nm) is well absorbed in the red pigment P.R. 22, whereas the absorption of the red pigment P.R. 122 is substantially low at 532 nm. These results [34] indicate, that tattoos containing yellow or red colours have unpredictably different response to laser therapy by using ruby laser and Nd:YAG laser. Therefore, it seems to be a hopeless attempt to correlate the colour of a tattoo and the laser wavelength for tattoo removal without any further information about the tattoo pigment used.

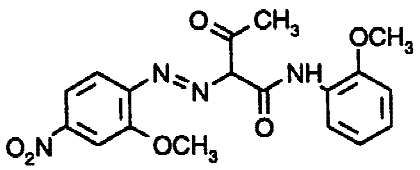
The TEM pictures of P.R. 122 indicate that the tattoo suppliers 1 and 2 have different sources for their pigment P.R. 122. Because of specific manufacturing procedures, pigments had aggregated in completely different shapes and sizes. Supposing that it is easier to destroy a thin needle than a large cubic particle by using Q-switched lasers, the outcome of therapy for the two specimens has to be different.

Moreover, the applied light intensity decreases rapidly during penetration into a pigment particle. The light intensity I_0 is reduced to I by using $I = I_0\, e^{-\alpha(\lambda)d}$, $\alpha(\lambda)$ is the absorption of the pigment, d the distance of light propagation. The absorption of light in the pigments is strong, comparable to metallic absorption. The phthalocyanine pigment P.B 15 exhibited an absorption $\alpha(\lambda = 694\ \text{nm})$ of $2 \times 10^5\ \text{cm}^{-1}$. If $d = 1/\alpha(\lambda)$, the light intensity I_0 is reduced to 37%, this value corresponds to a distance of about 50 nm of light propagation into the pigment. However, the size of the pigments ranges from a few nanometers to several hundred nanometers. After tattooing into skin, the pigment particles and agglomerates are exclusively found intracytoplasmatically, lying in membrane-bound structures, identified as secondary lysosomes [38]. This finding is due to active phagocytosis into dermal cells (macrophages, fibroblasts). The resulting pigment agglomerates range up to a few micrometers in diameter. Thus, with regard to pigments on the micrometer scale, the laser irradiation interacts mainly with the surface of the tattooed pigments.

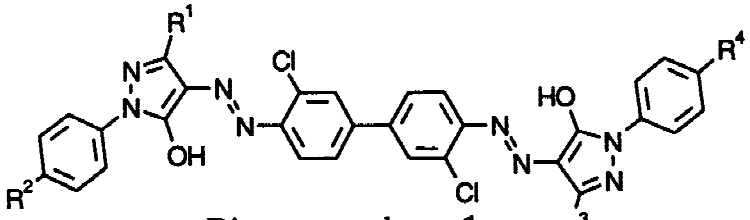
The goal of tattoo treatment by using Q-



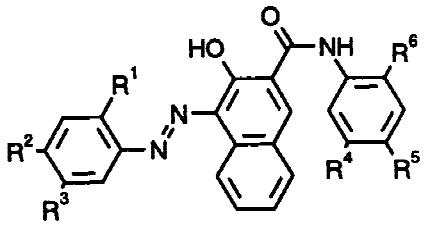
Disazodiarylides **1a** - **1d**



Monoazo Pigment **2**



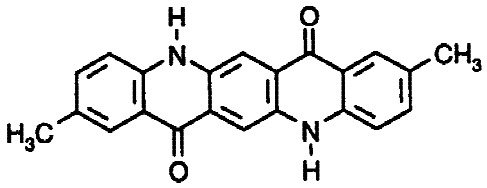
Disazopyrazolone **1e**



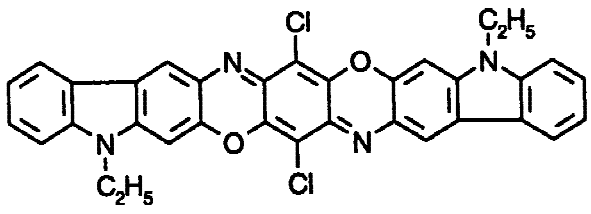
Naphthol AS Pigments **3a** - **3e**

Nr	R^1	R^2	R^3	R^4	R^5	R^6
1a	CH_3	H	H	H	H	CH_3
1b	H	CH_3	H	H	CH_3	H
1c	OCH_3	Cl	OCH_3	OCH_3	Cl	OCH_3
1d	OCH_3	H	OCH_3	OCH_3	H	OCH_3
1e	CH_3	H	CH_3	H	-	-

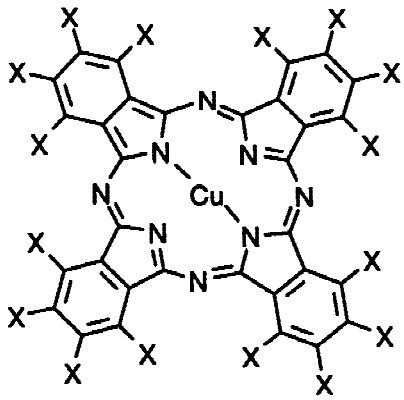
Nr	R^1	R^2	R^3	R^4	R^5	R^6
3a	OCH_3	H	SO_2NEt_2	Cl	OCH_3	OCH_3
3b	Cl	H	Cl	H	H	OCH_3
3c	CH_3	H	NO_2	H	H	H
3d	Cl	Cl	Cl	H	H	CH_3
3e	H	$CONH_2$	H	H	H	OC_2H_5



Quinacridone **4**



Dioxazine **5**



Cu - Phthalocyanines

6 (X = H)
7 (X = Cl)
8 (X = Cl, Br)

Fig. 1. Chemical structures of the pigments according to Tables 1 and 2.

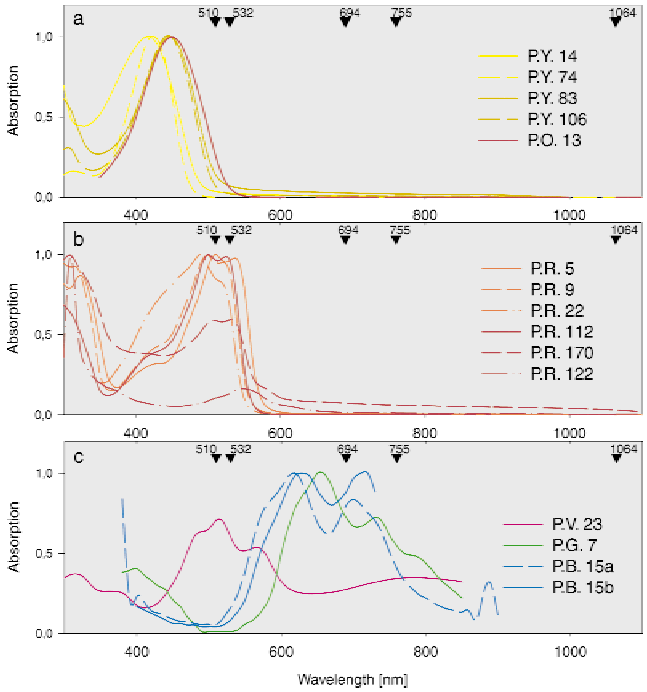


Fig. 2. **A:** The wavelength-dependent absorption $\varepsilon(\lambda)$ of yellow and orange pigments calculated with regard to the absorption maximum. The symbols inside the picture indicate the wavelength of Q-switched lasers used for tattoo removal. **B:** The wavelength-dependent absorption $\varepsilon(\lambda)$ of red pigments calculated with regard to the absorption maximum. The symbols inside the picture indicate the wavelength of Q-switched lasers used for tattoo removal. **C:** The wavelength-dependent absorption $\varepsilon(\lambda)$ of violet, blue, and green pigments calculated with regard to the absorption maximum. The symbols inside the picture indicate the wavelength of Q-switched lasers used for tattoo removal. The spectral range is restricted to 850 nm.

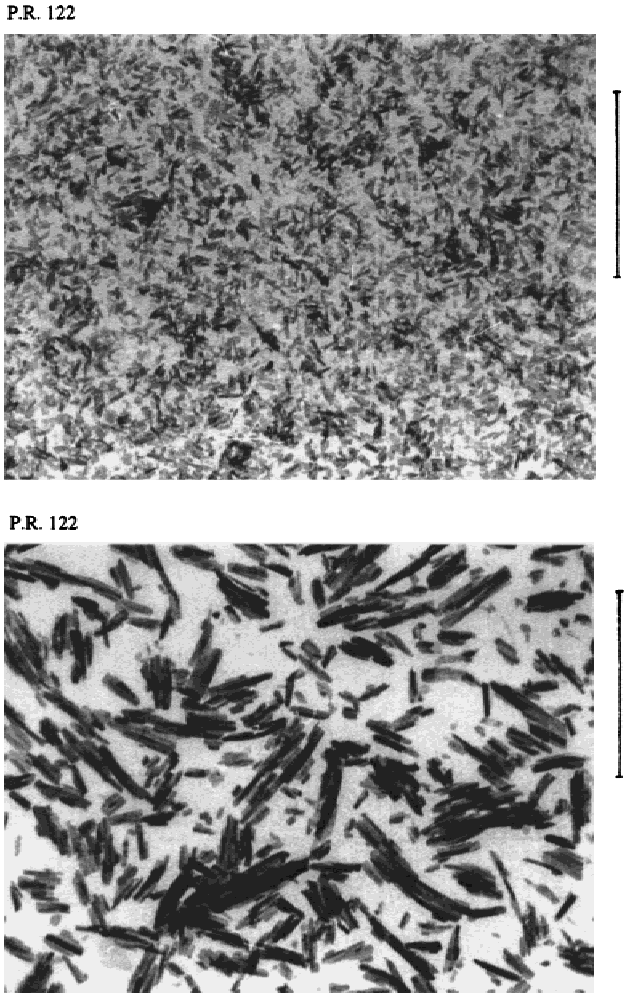


Fig. 3. Transmission electron microscopy images of two different shapes of the pigment P.R. 122. Scale bars = 1,000 nm.

switched lasers is to lighten the coloured areas of the skin, and different aspects must be discussed. The absorption of light pulses in the pigments is the first and important step to tattoo removal. After excitation by laser pulses, a part of absorbed energy is converted to heat (photothermal effect) or breaks chemical bonds inside the pigment (photochemical effects). After the ultrashort heating of the pigment surface by using a pulse duration of a few nanoseconds, a part of the energy is transported into the pigment by thermal conductivity and may lead to disruption of the pigment.

Similar findings are published by Watanabe et al. [39]. They investigated ultrastructural alterations of melanosomes in black guinea pig skin after exposure to ultrashort light pulses and provided evidence for the thermal initiation of melanosomal disruption.

At the same time, the extremely hot surface of the pigment may cause ultrashort heating up of a thin shell of surrounding tissue. The following expansion of this water-containing shell may induce a negative pressure and a shock wave near to the surface of the pigment. These shock waves may help to destroy the surrounding cellular structures and the tattooed compounds mechanically. Recently, an anomalous photoacoustic effect was reported when a suspension of small particles in water was irradiated by a Q-switched Nd:YAG laser (532 nm), where high-temperature reactions and gas expansions induced shock waves [40].

As a response, a multitude of mechanisms may occur at the same time. Large aggregates and agglomerates break down into smaller crystals. Particles pulverize and form a solution of pigment molecules. Molecules can break up, resulting in decomposition products or molecular structure change. Because of fragmentation of the tattoo particles as histologically already shown

[38], small pigment particles, unknown decomposition products, and newly generated chemical compounds then may be removed from the skin by means of lymphatic system. This mechanism could induce a decrease of the colour strength of the pigments responsible for a noticeable lightening of a coloured tattoo. However, there is also the possibility of laser-induced darkening of a tattoo caused by Fe_2O_3 in a cosmetic tattoo [41].

The adverse reactions regarding tattoos published in literature during the past few decades seemed to be induced mainly by inorganic pigments containing metals such as mercury, cadmium, chromium, cobalt, or iron. In addition, unknown admixtures and contaminations of the pigments have to be considered. Our study indicates that, in coloured tattoos, industrial organic pigments are replacing the inorganic metal-containing pigments to a large extent.

Because the laser energy is converted to heat, one has to take into account that diarylide pigments (P.Y. 13, 14, 17, 83, or P.O. 13) undergo cleavage at temperatures above 200°C, yielding a mixture of monoazo dyes and primary aromatic amines. Aromatic amines are compounds that have been shown to be or are suspected of being carcinogenic [42]. Amines may react to the cancerogenic nitrosamines or provoke allergic and asthmatic reactions. Increasing the temperature above 280°C leads to 3,3′-dichlorobenzidine, a proven animal carcinogen [43].

Thus, the users of Q-switched lasers should keep in mind that the question of whether the laser therapy of tattoos presents a toxicologic or cancerogenic risk cannot be definitely answered today. The first reports on adverse reactions after tattoo treatment by using Q-switched lasers [44,45] may confirm our suggestions. Therefore, we are performing further investigations regarding the analysis of possible decomposition products.

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